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The Use of Infrared Reflectance Spectroscopy for the Automatic Continuous Monitoring and Control of Whey Ultrafiltration Processes

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ABSTRACT

A system, based on the infrared reflectance spectroscopic method for the rapid, simultaneous determination of protein, fat, carbohydrate, nonprotein nitrogen and total solids in milk and liquid dairy products, has been developed for continuous analysis of liquid streams for those components. These analyses are used to predict and to control the composition of the whey protein concentrate during ultrafiltration. The ratio of protein to total solids in the concentrate is controlled automatically by a valve in the product outlet line which is adjusted by feedback of the analytical data. The concentration data have also been used to provide alarm systems for the measurement of deterioration, or total failure, of the ultrafiltration membranes. The analytical system has been designed to operate on a whey ultrafiltration plant in continuous operation, with automatic sampling, relevant calibration and CIP (cleaning in place), so that it can be operated with minimum supervision.

The whey ultrafiltration process is an example of many processes that, with little modification of the analytical instruments, can be monitored and controlled. Thus concentration of ethanol, CO_2 or of some specific compound, can be measured and used to control many other processes including fermentation and hydrogenation.

INTRODUCTION

The complexity of food samples and legislation affecting the levels of ingredients and the labelling of products has increased to the extent that analytical methods have had to be developed which enable routine and automatic analysis. Furthermore, increasing raw material costs has made it is necessary to avoid wastage by adhering closely to specifications for manufactured foods.

Several traditional analytical methods, e.g. Kjeldahl for protein and solvent extraction for fat, have been successfully automated and special automatic methods have been developed (Hitchcock, 1983), e.g. density, gas–liquid chromatography and liquid chromatography for fat, nuclear magnetic resonance for protein and liquid chromatography for carbohydrates. The major drawback of these techniques is that they require trained laboratory operators and a particular piece of equipment for each class of compound; they are therefore costly to operate. Moreover, since they cannot readily be adapted to in-line analysis, delays between taking samples and receiving the compositions make adjustment of the process to maintain product composition slow and often wasteful.

In order to develop a process control system for the food industry it is essential to use a method which can simultaneously analyse a number of components in a single analyser preferably in such a way as to cause no contamination problems in the product and which can be operated by plant operatives rather than laboratory personnel.

The development of an infrared reflectance spectroscopic method for the simultaneous determination of protein, fat, nonprotein nitrogen, carbohydrate and total solids in milk and liquid dairy products (Kennedy *et al.*, 1985) has provided an analytical system which is capable of satisfying the analytical criteria of a fully automated process control system. We now report the development of this analytical system into a fully automated system for the control of whey ultrafiltration processes, with control based on the protein to total solids ratio to provide feedback

to the process control valve. A process flow cell of improved sensitivity has been incorporated into the laboratory analyser, the analyser being linked to a microcomputer which provides the necessary data manipulation for process control, data output, automatic washing and zero adjustment of the analyser and, with the aid of preset values, a system of monitoring of the condition of ultrafiltration membranes with inbuilt alarm facilities. Design of the installation position has also been undertaken to ensure optimum control of the system, with minimum delay times, and to ensure freedom from contamination of food products.

EXPERIMENTAL

Materials

All samples used to calibrate the analyser were obtained from a whey ultrafiltration plant and accurate analytical data, obtained via traditional wet chemical methods, namely Kjeldahl (Lillevik, 1970) for protein and nonprotein nitrogen, Rose-Gottlieb (Hubbard *et al.*, 1977) for fat and Lane & Eynon (1923) for carbohydrate with total solids content being determined from the weight of dried material taken. Compositions of these powders are given in Table 1. All samples were dissolved by gently stirring the required weight, portionwise, in distilled water and diluting the slurry to 500 ml with distilled water.

Equipment

For this study a Miran-80 Computing Quantitative analyser, fitted with a M425 process flow-through cell (volume 12·1 ml) and cell-thermostatted heater was used. This instrument has now been superseded by the Miran-980 analyser (Buck Scientific Ltd) which has an improved microprocessor and the ability to compute its own calibration (P) matrix. A simple flow system was devised for laboratory simulation, using a 100 ml/min peristaltic pump.

Calibration of analyser

The Miran-80 analyser was programmed to read absorbance values at the wavelengths shown in Table 2 and, after zeroing the analyser with water,

TABLE 1
Composition of Plant-Derived Material Used to Prepare Calibration Solutions

Sample no.	*Composition of powder sample (% w/w)*						*Wt taken[a] per 500 ml*
	Fat	*Total protein*	*Nonprotein nitrogen*	*Lactose*	*Moisture*	*Ash*	
1	9·44	76·6	4·11	1·9	4·60	2·49	110
2	7·02	56·0	5·09	24·0	6·60	3·73	81
3	4·07	40·4	4·47	45·6	3·17	4·24	61
4	4·25	39·9	4·29	46·2	3·81	5·49	58
5	4·11	38·6	4·47	47·5	3·64	5·13	55
6	4·63	37·1	3·57	47·3	n/d	n/d	51
7	4·23	37·0	4·11	52·7	4·20	4·28	50
8	3·94	35·5	3·93	48·9	3·31	4·61	48
9	4·80	35·3	3·93	46·8	4·31	5·02	45
10	4·53	33·2	4·47	52·5	4·53	5·40	42
11	3·71	30·9	4·47	52·5	3·35	5·81	40
12	0·29	2·92	2·92	83·4	n/d	n/d	27

[a] These solutions were used to obtain the data in Tables 4 to 8.
n/d = not determined.

TABLE 2
Analytical Wavelengths and Designation Used

Wavelength (10^{-6} m)	*Designation*
3·97	Reference
5·74	Fat
6·35	Total protein
6·68	Nonprotein nitrogen[a]
8·25	Total solids
8·80	Nonprotein nitrogen[b]
9·54	Carbohydrate

[a] Value used for modified five-component analysis system.
[b] Value used for initial five-component analysis system.

each solution was analysed in turn by pumping the solution for *ca.* 2 min to ensure a representative sample was present in the flow cell. Throughout all tests the analysis was performed on static samples (zero flow through the cell). Each sample was analysed twice (with a short pumping time between each analysis) before the system was flushed with water and the next sample introduced. Three sets of absorbance values were recorded for each of the two aliquots of each sample and the arithmetic mean of the six results was calculated to obtain a representative absorbance for each wavelength.

Using these results and the known concentrations of the components in each sample, four different calibration (P) matrices were derived with the aid of a laboratory microcomputer. (The newer Miran-980 analyser is programmed to do this, dispensing with the need for the matrix manipulation program and laboratory microcomputer.) The different P matrices arose from the choice of two different five-component analyses using different detection wavelengths for nonprotein nitrogen, each using either a zero or nonzero intercept to the calibration response.

Analysis of samples using the initial five-component analysis system

Once the analyser was programmed with the P matrix each solution was re-analysed as described for the initial calibration, with three sets of absorbance values for each of two aliquots being recorded for each sample, the analyzer being zeroed on water between samples.

Adsorption of material by the flow-cell

In order to test whether the materials used to make the process flow-through cell had any affinity for components of the samples, one sample (sample 4) was allowed to stand, after analysis, in the cell (with zero flow) for 2 h prior to being re-analysed.

A further test for adsorption of material on the MIR crystal was made by pumping water through the flow-through cell at 200 ml/min whilst monitoring the analysis results.

Stability of the analyser

The stability of the analyser was assessed by thermostatting the flow-through cell at 26 °C and pumping the sample overnight with analyses

being performed (with positive flow through the cell) *ca.* every 17 min. Ambient temperature was 22 °C at the start and 19 °C at the end of the test. The initial flow rate was set at 150 ml/min but due to wear in the pump tubing this rose during the experiment to 251 ml/min.

Effect of rapid changes in composition

The effect of a rapid change in stream composition was studied by continuously analysing one sample flowing through the analyser and then rapidly switching the inlet tube to a second solution (compositions of these solutions are given in Table 3) with the minimum of disruption to flow. Analytical results were recorded until new, stable values were obtained, with the analyser adjusted to provide one complete, five-component analysis every 2 min. The flow rate was maintained at 100 ml/min throughout.

TABLE 3
Synthetic Solutions Used for Change-Over Tests

Calculated concentration of components[a] (% *w/v*)				
Test 1	*Test 2*	*Test 3*	*Test 4*	*Test 5*
Permeate[b]	Permeate[b]	Retentate[b]	Retentate[b]	P:R[c] = 10:90
0·0	0·0	1·9	1·9	1·8
0·2	0·2	8·1	8·1	7·3
5·5	5·5	15·7	15·7	14·6
0·0	0·0	0·4	0·4	0·4
4·6	4·6	3·8	3·8	3·8
		change-over		
P:R[c] = 98:2	P:R[c] = 90:10	P:R[c] = 2:98	P:R[c] = 10:90	Permeate[b]
0·1	0·1	1·9	1·8	0·0
0·2	0·8	7·9	7·3	0·2
5·7	6·5	15·5	14·7	5·5
0·0	0·1	0·4	0·4	0·0
4·6	4·5	3·8	3·8	4·6

[a] Order of results shown in each column: fat, total protein, total solids, nonprotein nitrogen, and carbohydrate.
[b] The permeate and retentate powder samples used for this study were not analysed by non-infrared techniques.
[c] P:R = permeate to retentate ratio.

RESULTS AND DISCUSSION

Calibration

Our recent paper (Kennedy *et al.*, 1985) dealing with the calibration of a laboratory version of the Miran-80 reflectance infrared computing quantitative analyser for analysis of liquid dairy products indicated that a compromise between maximum information and accuracy had to be made. However, the configuration of the process flow-through cell is such that almost twice the surface area of MIR crystal is in contact with sample, which flows over both faces of the crystal rather than lying on one surface only as is the case with the laboratory version of the analyser. This results in higher energy levels and an improved signal to noise ratio. It was therefore decided to re-investigate both the initial and modified five-component analysis system using the process flow-through cell to obtain the best system for calibration. The mean absorbance values of solutions prepared from the weights of powder shown in Table 1 dissolved in 500 ml water at the chosen wavelengths were entered into a laboratory computer which was programmed to calculate the predicted results using both zero and nonzero intercepts for calibration for the initial method (using $8{\cdot}80 \times 10^{-6}$ m for nonprotein nitrogen) and the modified method (using $6{\cdot}68 \times 10^{-6}$ m for nonprotein nitrogen) using a system of matrix manipulation. The results are shown in Tables 4 to 8.

The computer-predicted results for fat (Table 4) and nonprotein nitrogen (Table 6) show that when the instrument is accurately calibrated with material only from the plant process (i.e. not using synthetic samples or nonwhey products) good correlation can be obtained down to the 0·25% level. In all cases, the computer-predicted results indicated very little difference between the four possible calibration systems unlike the laboratory-based system (Kennedy *et al.*, 1985) which indicated that the modified system was superior. Because the noise levels on absorbances were lower at $8{\cdot}80 \times 10^{-6}$ m than at $6{\cdot}68 \times 10^{-6}$ m, the initial method for a five-component analysis system was chosen and used with a nonzero intercept.

Analysis of process materials

The P matrix for the initial method with nonzero intercept was calculated and programmed into the Miran-80 analyser (via its keyboard) and used to re-analyse all twelve samples. One set of triplicate concentration values for

TABLE 4
Comparison of Computer-Predicted Concentrations with Calculated Values for Fat

Sample no.	*Concentration of fat (% w/v)*				
	Calculated	*Initial method*		*Modified method*	
		Zero intercept	*Nonzero intercept*	*Zero intercept*	*Nonzero intercept*
1	1·99	2·00	1·99	2·00	2·00
2	1·06	1·02	1·04	1·02	1·03
3	0·48	0·48	0·46	0·47	0·45
4	0·47	0·52	0·51	0·52	0·52
5	0·43	0·44	0·43	0·45	0·45
6	0·47[a]	0·44	0·43	0·42	0·41
7	0·41	0·42	0·40	0·43	0·43
8	0·37	0·39	0·39	0·38	0·38
9	0·41	0·36	0·36	0·37	0·37
10	0·36	0·35	0·36	0·33	0·33
11	0·29	0·26	0·27	0·27	0·28
12	0·01[a]	0·02	0·03	0·04	0·05

[a] No correction for moisture content.

TABLE 5
Comparison of Computer-Predicted Concentrations with Calculated Values for Total Protein

Sample no.	*Concentration of total protein (% w/v)*				
	Calculated	*Initial method*		*Modified method*	
		Zero intercept	*Nonzero intercept*	*Zero intercept*	*Nonzero intercept*
1	15·7	15·6	15·6	15·6	15·7
2	8·47	8·64	8·47	8·66	8·47
3	4·77	4·38	4·53	4·34	4·56
4	4·45	4·65	4·74	4·65	4·75
5	4·09	4·03	4·13	4·09	4·13
6	3·78[a]	3·73	3·75	3·62	3·67
7	3·55	3·37	3·51	3·41	3·58
8	3·30	3·40	3·32	3·35	3·26
9	3·04	3·04	3·03	3·09	3·07
10	2·66	2·81	2·69	2·67	2·56
11	2·39	2·31	2·17	2·37	2·20
12	0·16[a]	0·39	0·27	0·50	0·34

[a] No correction for moisture content.

TABLE 6

Comparison of Computer-Predicted Concentrations with Calculated Values for Nonprotein Nitrogen

Sample no.	*Concentration of nonprotein nitrogen (% w/v)*				
	Calculated	*Initial method*		*Modified method*	
		Zero intercept	*Nonzero intercept*	*Zero intercept*	*Nonzero intercept*
1	0·86	0·87	0·87	0·87	0·87
2	0·77	0·71	0·73	0·71	0·73
3	0·53	0·51	0·49	0·52	0·50
4	0·48	0·53	0·52	0·52	0·52
5	0·47	0·47	0·46	0·46	0·45
6	0·36[a]	0·38	0·37	0·37	0·36
7	0·39	0·37	0·35	0·38	0·36
8	0·36	0·41	0·42	0·40	0·41
9	0·34	0·36	0·36	0·36	0·36
10	0·36	0·30	0·31	0·29	0·29
11	0·35	0·32	0·34	0·33	0·34
12	0·16[a]	0·15	0·16	0·16	0·17

[a] No correction for moisture content.

TABLE 7

Comparison of Computer-Predicted Concentrations with Calculated Values for Total Solids

Sample no.	*Concentration of total solids (% w/v)*				
	Calculated	*Initial method*		*Modified method*	
		Zero intercept	*Nonzero intercept*	*Zero intercept*	*Nonzero intercept*
1	21·0	21·1	21·1	21·1	21·1
2	15·1	14·8	14·9	14·9	14·1
3	11·8	11·7	11·6	11·8	11·7
4	11·2	11·5	11·5	11·5	11·5
5	10·6	10·9	10·8	10·8	10·8
6	10·2[a]	9·97	9·96	9·82	9·81
7	9·58	9·32	9·26	9·42	9·40
8	9·28	9·34	9·38	9·27	9·28
9	8·61	8·71	8·71	8·79	8·79
10	8·02	8·08	8·13	7·89	7·90
11	7·73	7·59	7·65	7·67	7·69
12	5·40[a]	5·40	5·45	5·57	5·59

[a] No correction for moisture content.

TABLE 8
Comparison of Computer-Predicted Concentrations with Calculated Values for Lactose

Sample no.	*Concentration of lactose (%w/v)*				
	Calculated	*Initial method*		*Modified method*	
		Zero intercept	*Nonzero intercept*	*Zero intercept*	*Nonzero intercept*
1	0·40	0·52	0·52	0·51	0·51
2	3·63	3·28	3·29	3·32	3·31
3	5·38	5·50	5·49	5·57	5·57
4	5·15	5·14	5·13	5·12	5·12
5	5·03	5·16	5·16	5·07	5·07
6	4·82[a]	4·71	4·70	4·66	4·66
7	5·05	4·61	4·61	4·66	4·66
8	4·54	4·62	4·63	4·60	4·60
9	4·03	4·40	4·40	4·43	4·43
10	4·21	4·18	4·19	4·13	4·13
11	4·06	4·28	4·29	4·29	4·29
12	4·50[a]	4·32	4·33	4·38	4·38

[a] No correction for moisture content.

each sample is shown in Table 9. The variation between triplicate analyses is, in most cases, 0·2% w/v or less which compares very favourably with the reproducibility obtainable with the traditional wet chemical methods. A comparison with the calculated values for each solution (comparing Table 9 with Tables 4 to 8) shows that very good correlation is achieved and conversion factors to equate the infrared method with traditional methods are not required as the method of calibration of the instrument automatically takes these into account.

It should be noted that a calibration matrix for the modified method could also be programmed into the Miran-80 analyser and, because of the higher signal to noise ratio for the process flow-through cell, there were no strong nondiagonal properties in the P matrix and the Miran-80 analyser was able to convert absorbance values into concentration values with none of the difficulties encountered (Kennedy *et al.*, 1985) with the laboratory version of the analyser. Thus the alternative system can be used, if particular parameters in a process, such as a by-product having an interfering infrared absorption at $8{\cdot}80 \times 10^{-6}$ m, prevent adoption of the initial method.

TABLE 9

Triplicate Concentration Values Obtained on Re-analysing the Solutions After Calibration of the Analyser

Sample no.	*Concentration (%)*				
	Fat	*Total protein*	*Total solids*	*Nonprotein nitrogen*	*Lactose*
1	1·8	15·0	20·0	0·9	0·3
	1·9	15·5	20·6	0·9	0·7
	1·9	15·3	20·4	0·9	0·6
2	0·9	8·0	14·2	0·7	3·5
	0·9	7·9	14·1	0·6	3·5
	0·9	7·9	14·2	0·6	3·5
3	0·4	4·4	11·5	0·6	5·3
	0·4	4·6	11·9	0·6	5·5
	0·5	4·5	11·6	0·6	5·2
4	0·6	5·2	12·2	0·6	4·8
	0·6	5·2	12·3	0·7	5·1
	0·6	5·3	12·3	0·6	4·8
5	0·3	4·1	10·7	0·5	4·8
	0·4	4·1	10·7	0·5	4·9
	0·5	4·3	11·0	0·5	4·8
6	0·5	4·1	10·2	0·6	4·2
	0·5	3·9	10·0	0·6	4·0
	0·6	4·2	10·3	0·6	4·0
7	0·6	4·3	10·5	0·5	4·2
	0·6	4·3	10·5	0·5	4·1
	0·6	4·2	10·5	0·5	4·2
8	0·5	3·4	9·5	0·6	3·9
	0·6	3·6	9·8	0·5	4·2
	0·5	3·5	9·6	0·6	3·9
9	0·4	3·7	9·8	0·6	4·1
	0·4	3·6	9·7	0·5	4·2
	0·4	3·6	9·8	0·6	4·2
10	0·5	3·1	8·7	0·5	3·4
	0·5	3·3	8·9	0·5	3·6
	0.5	3·3	9·0	0·5	3·5
11	0·3	2·7	8·7	0·5	4·1
	0·4	2·6	8·3	0·6	3·8
	0·5	2·6	8·4	0·6	3·8
12	0·2	0·5	5·5	0·3	3·4
	0·2	0·4	5·7	0·3	3·7
	0·2	0·4	5·7	0·3	3·6

Interference by adsorption

The test carried out to identify specific adsorption of any component of the sample by allowing the sample to remain in contact with the cell (with zero flow) for 2 h between successive analyses resulted in no change in concentration values outside the limits of variation found between successive duplicate analyses. This, together with the results from pumping water through the cell between samples, which gave values of 0·1 or zero for all components each time, confirms that, if any material is adsorbed by the MIR crystal or construction materials in the flow-through cell, it is of such a small amount as to be insignificant for analytical purposes. However, for production use it would be advisable to flush the cell periodically with a detergent solution to prevent any build-up of material and to avoid bacterial contamination.

Long-term stability

Measurement of absorbance during an overnight test (Table 10) indicates that changes occur, but by including a reference wavelength ($3{\cdot}97 \times 10^{-6}$ m) the changes due to flow-rate and temperature can be minimised (even the large variations found for fat which are the result of interference from small changes in the adjacent water resonance) such that the concentration values (Table 11) recorded over the 15 h period do not show any significant changes. Since the MIR technique is very

TABLE 10
Variation in Absorbance Values with Time for Overnight Stability Test

Analysis no.	*Approx. time*	*Reference*	*Absorbances measured*				
			Fat	*Total protein*	*Total solids*	*Nonprotein nitrogen*	*Lactose*
1	6.0 pm	0·0004	−0·0057	0·0810	0·0424	0·0657	0·1623
11	9.0 pm	0·0006	−0·0076	0·0757	0·0421	0·0681	0·1642
21	12.0 pm	0·0002	−0·0091	0·0763	0·0424	0·0677	0·1639
31	3.0 am	0·0014	−0·0111	0·0739	0·0422	0·0664	0·1635
41	6.0 am	0·0017	−0·0117	0·0753	0·0430	0·0668	0·1628
52	9.0 am	0·0017	−0·0104	0·0752	0·0429	0·0679	0·1671

TABLE 11
Variation in Concentration Values with Time for Overnight Stability Test

Analysis no.	*Approx. time*	*Concentration (% w/v)*				
		Fat	*Total protein*	*Total solids*	*Nonprotein nitrogen*	*Lactose*
1	6.0 pm	0·6	5·2	12·2	0·6	5·0
11	9.0 pm	0·6	5·2	12·5	0·6	5·1
21	12.0 pm	0·6	5·2	12·5	0·6	5·2
31	3.0 am	0·6	5·1	12·4	0·6	5·2
41	6.0 am	0·6	5·1	12·5	0·6	5·4
52	9.0 am	0·6	5·2	12·6	0·5	5·4

sensitive to changes in refractive index, it is imperative that the flow-through cell is thermostatted and that all analyses should be made with zero flow through the cell to combat refractive index changes due to temperature and pressure, respectively. It is also considered appropriate to include an automated wash/re-zeroing facility into the overall production control program.

Response to rapid changes

In order to detect the speed with which the analyser can respond to compositional changes in the process stream, the sample inlet was rapidly switched between samples of similar and different compositions (see Table 3) to simulate slight changes in composition due to nonequilibration of the process and large changes due to failure of an ultrafiltration membrane. From a comparison of the analysis immediately prior to the change-over with the second and third analyses after the change-over, it can be seen (see Table 12) that the concentration values normally stabilise by the second analysis although for very large changes in composition (e.g. Test 5) one more analysis was required. This shows that the analyser is capable of responding to any change of concentration in about 2 min under the conditions used in this simulated system. Obviously the rate at which the analyser will respond when installed in a production process will depend on the dead volume of the associated pipework between the ultrafiltration membranes and the analyser and for rapid control this must be kept to a minimum by the use of fast-flowing sample loops and small-bore sample lines.

TABLE 12
Effect of Rapid Changes in Stream Composition on Concentration Values Recorded by the Analyser

Concentration of components[a] (% *w/v*)				
Test 1	*Test 2*	*Test 3*	*Test 4*	*Test 5*
0·1	0·1	2·0	1·9	1·9
0·0	0·0	7·8	7·9	7·5
5·1	5·2	15·6	15·7	14·7
0·0	0·0	0·0	0·4	0·5
4·6	4·6	3·8	4·0	4·0
0·1	0·1	1·9	1·8	2·0
0·0	0·0	8·1	7·9	7·4
5·6	5·4	15·7	15·7	14·6
0·0	0·0	0·4	0·5	0·3
4·5	4·6	3·8	3·8	3·9
— — — — — — — —		stream change		— — — — — — —
0·2	0·2	1·7	1·9	0·7
0·0	0·5	8·9	7·4	2·0
5·9	6·5	16·4	16·0	7·2
0·0	0·0	0·7	0·7	0·0
4·8	4·7	3·7	4·0	4·0
0·1	0·1	2·0	1·9	0·4
0·0	0·8	8·2	7·7	0·5
5·8	6·5	15·8	16·4	5·2
0·0	0·0	0·2	0·9	0·0
4·7	4·5	3·8	3·9	4·4
0·1	0·1	2·1	2·0	0·2
0·1	0·6	7·9	7·6	0·2
5·8	6·3	15·7	14·9	5·5
0·0	0·0	0·3	0·6	0·0
4·7	4·6	3·9	4·0	4·5

[a] Order of results shown in each column: fat, total protein, total solids, nonprotein nitrogen, and carbohydrate.

AUTOMATED CONTROL SYSTEM DESIGN

The foregoing results show that the process control version of the analyser can provide the rapid, simultaneous determination of protein, fat, carbohydrate, nonprotein, nitrogen and total solids in liquid streams without the need for manual input of samples. Thus the analyser satisfies the basic criteria for the fully automated control of whey ultrafiltration processes. By coupling this analyser to a microcomputer which is also

linked to liquid stream flow meters and the process control valve, total automated control of the process can be achieved using the parameter

$$\frac{\text{total protein} - \text{nonprotein nitrogen}}{\text{total solids}}$$

which is calculated by the microcomputer to control the process control valves. This parameter is chosen for the ultrafiltration process to ensure that material of correct composition (in terms of percent w/w protein after drying) is allowed to flow to the subsequent drying process. Thus by selection of such a parameter a basic composition of the final product can be obtained and when the input from flowmeters, clock, etc., is used in the computations, the total output and individual component compositions and throughput can be determined for any automated process.

By selecting the necessary upper and lower limit control values, the microcomputer will be able to provide alarm facilities to indicate deterioration or total failure of the ultrafiltration membranes via a steady increase, or sudden vast increase, respectively, in the protein content of the permeate stream. This alarm facility can be most effectively monitored using a second analyser sampling the permeate line only, which will also provide accurate carbohydrate concentration for any subsequent fermentation process, e.g. the conversion of whey permeate to alcohol (Mehnia & Cheryan, 1984). However, a system of stream sharing on a single analyser can provide a similar facility (see Fig. 1) albeit with some loss of monitoring of the separate streams and a delay in analysis times due to the need to ensure equilibration of sample in the flow-through cell. The data output from the microcomputer can be designed to provide full documentation of production output, etc., which will further reduce costs.

The design of the sampling facilities in the process flow system is critical if the delay between producing a particular sample and analysing it is to be kept to a minimum and thus prevent quantities of materials outside the specification being produced. To satisfy this, it is recommended that fast-flowing sample loops be used for the rapid transfer of sample from the point of production to the analysers, with narrow bore take-off points adjacent to the analyser to facilitate sampling when required (Fig. 1). For a typical application the whey input flow rate varies between 600 and 150 litres/min (depending on membrane condition, product, etc.) with the product flow rates being between 100 and 5 litres/min for the retentate stream in a 2·5 in (6·6 cm) diameter pipe and between 500 and 145 litres/min for the permeate stream in a 6 in (15·2 cm) diameter pipe. For fast sample

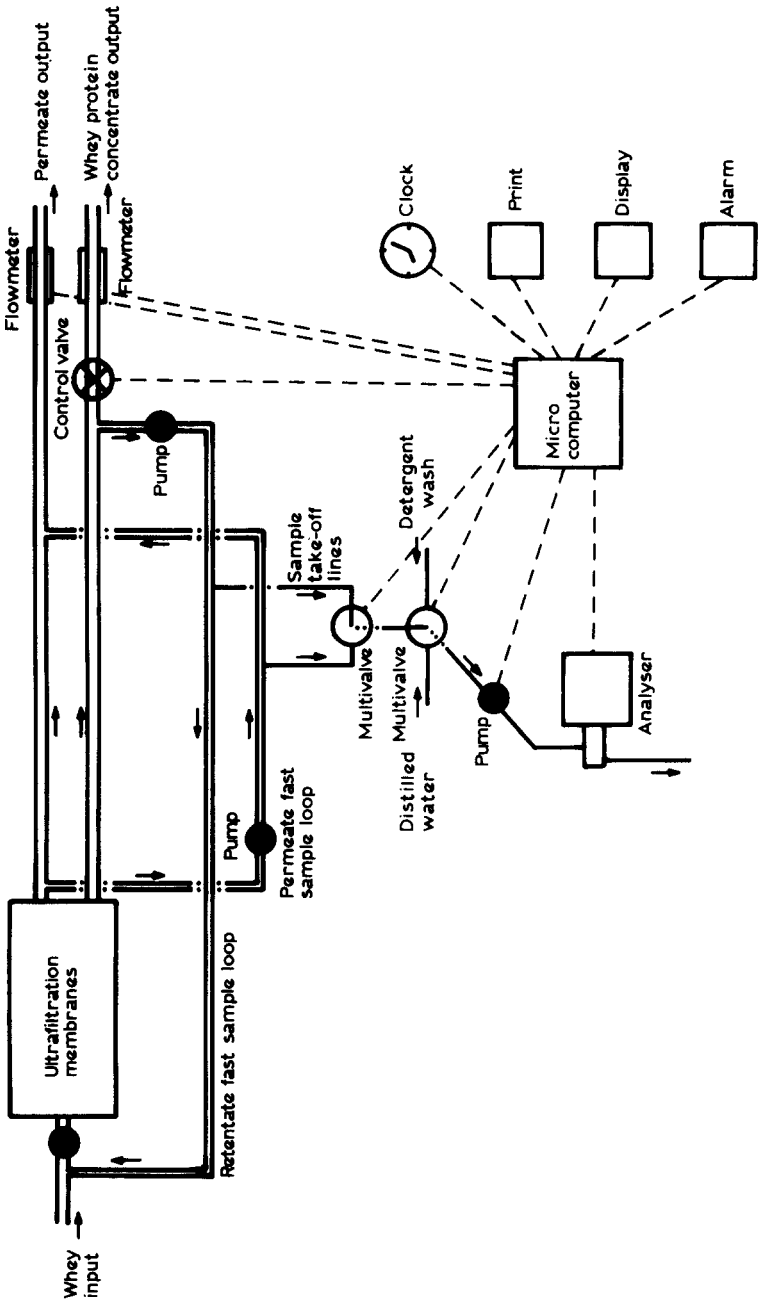

Fig. 1. Schematic representation of the whey ultrafiltration process control system.

loops of 0·5 in (1·3 cm) diameter pumped at 3 litres/min the delay time for a distance of 100 ft (30·5 m) between the sample point and the narrow bore take-off point will be less than 2 min. If the sample only flows at 100 ml/min via 0·25 in (0·6 cm) diameter tubing through the analyser when required, the minimum of sample is lost or, if returned to the production system, the amount of contamination will be minimal. The actual positions of the inlets and outlets of these fast sample loops are critical to ensure that: no closed loop situation can be set up; thorough mixing of remixed streams occurs prior to any re-analysis; the flow meters record material passed to subsequent processes; and no bypass of the process control valve exists.

By use of computer-controlled multivalves, the stream passing through the analyser can be selected, on demand, to be from the retentate or permeate fast sample loops, detergent wash solutions or distilled water to allow the analyser(s) to be re-zeroed automatically.

CONCLUSION

The laboratory-based system for analysis of milk products (Kennedy *et al.*, 1985) has been adapted for use as a continuous process liquid stream analyser which can be incorporated into a fully automated, computer-controlled process with feedback control of an industrial process, which can be operated adjacent to the production plant by plant operatives. Whilst this paper discusses ultrafiltration of whey, the system can readily be modified, by selection of the appropriate absorbance frequencies and control parameters, to provide a continuous fully automated control system for many other industrial processes, such as fermentations (using carbon dioxide, ethanol or a specific product as a control facility), hydrogenations (using starting material loss or product increase as control facility), etc. Existing methods for the monitoring of such reaction processes at present rely on lengthy extraction procedures (Varma *et al.*, 1984) prior to analysis by, for example, gas chromatography. Adoption of an infrared analysis system, as described in this paper, could overcome such problems.

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